Image Analysis of Iron Oxides under the Optical Microscope.

E.Pirard & S.Lebichot
Mineral Georesources and Geo-Imaging Group, GeomaC Department, Université de Liège, Liège, Belgium

ABSTRACT: Many mineralogists have disregarded reflected light microscopy for automated mineral identification since the advent of electron microscopy and particularly EDX microanalysis systems. Although obviously such systems are capable of resolving delicate mineral identification questions they suffer from high operating costs and low image acquisition rates. An additional reason for having disregarded optical microscopy in quantitative ore mineralogy during the last decades is that poor results have been obtained so far because of a very careless attention to the image acquisition protocol. In this paper, we demonstrate how taking advantage of all available information under the optical microscope may improve the identification of some important minerals. By using several interference filters and by rotating the polarizer / analyzer filter set, we obtain a stack of multispectral and multiradial images that can be segmented using multivariate discrimination techniques. In such a way, absolute reflectance, pleochroïsm, bireflectance and optical anisotropy contribute to the optimal classification of individual pixels into the user-defined mineral species that form the paragenesis under study. Preliminary results are shown on some parageneses where backscattered electron imaging typically proves to be poorly discriminating or useless: Hematite-Magnetite-Goethite in Banded Iron Formations; Ilmenite-Magnetite-Hematite in Magmatic-Ti ores; etc.

1 APPLIED MINERALOGY OF IRON ORES.

The mineralogy of iron ores, particularly those of sedimentary origin, is extremely simple and only involves three major iron-bearing mineral species: Hematite (Fe2O3), Magnetite (Fe3O4) and Goethite (FeO.OH), not to mention the more or less hydrated mineral mixtures designated under the generic but confusing term of Limonite. The traditional trading of iron ores is based on chemical specifications and size distribution requirements. However, in recent years, detailed studies of iron ores and their behavior in steelmaking processes have drawn attention on the need for a more detailed description of the microtexture of iron ores (Vieira et al., 2003; Clout, 2003). Of particular interest are the size distribution of hematite crystals (microplaty, recrystallised, granular,…), the mineral intergrowth (martite, hematite/goethite,…) and the microscopic arrangement of crystals (microporosity, …). In order to perform such a detailed analysis, mining and steelmaking companies have no alternative to the tedious visual inspection of thousands of grains either under the scanning electron microscope or the reflected light microscope. Despite spectacular progress in instrumentation (Sutherland & Gottlieb, 1991) the automatic identification of minerals under the microscope and the consequent description of microtextural arrangements still stumble over difficulties. This is particularly true for iron ores where all major minerals display poor contrast in their physical / chemical properties (table 1).

Mineral	R_{470}	R_{546}	R_{650}	Fe % wt	$\overline{Z}$ mean atom.nb.
Magnetite	20.9	20.7	20.7	72	21
Hematite	32.5/28.3	31.0/26.8	26.6/23.3	70	20,1
Goethite	17.2/19.9	15.5/17.5	14.4/16.1	63	19,2
Limonite				~= 60	~= 18,7
Ilmenite	19.7/16.9	19.2/16.4	20.2/18.0	37	19
Pyrrothite	32.1/37.0	36.3/41.4	41.5/45.4	64	22,4
Marcasite	44.8/51.6	49.1/56.2	48.3/53.4	47	20,7
Pyrite	45.6	51.7	54.4	47	20,7
Pentlandite	41.9	48.9	53.4	39	~= 23,4

Table 1: Reflectances, composition and average atomic number of major iron oxides and iron sulphides (from Jones, 1987; Criddle A. and Stanley J., 1993).

2 PRINCIPLES OF QUANTITATIVE ORE IMAGING

Digital imaging of mineral assemblages under the microscope can be achieved in several ways: either by using scientific grade CCD cameras or by scanning a scene with an electron beam and digitizing the output of a Robinson Backscattered Electrons detector (BSE imaging) or an X-Ray detector (EDX imaging). When high spatial resolution is a strong requirement, optical imaging cannot compete with scanning electron microscopy. However, in many cases, as with iron ores, optical imaging is a cheap and flexible alternative solution.

The widespread availability of scanning electron microscopes has attracted a lot of attention and impeded the development of research work on a rigorous visible light spectrometric imaging in optical microscopy. This lack of interest has been reinforced by the poor results obtained when trying to perform image analysis using color video or still video cameras. Considering the comprehensive measurement and compilation of visible specular reflectance curves published by Criddle and Stanley (1993) on behalf of the Commission for Ore Microscopy (IMA-COM), it appears worth attempting to reconcile both spectroscopy and imaging modes. This is only possible by using multispectral imaging and a proper calibration procedure as proposed in Pirard (2004). Fig. 1 shows how the use of narrow bandwidth (10nm) interference filters improves discrimination between Pyrite and Pentlandite as compared to the tri-stimulus color filters.

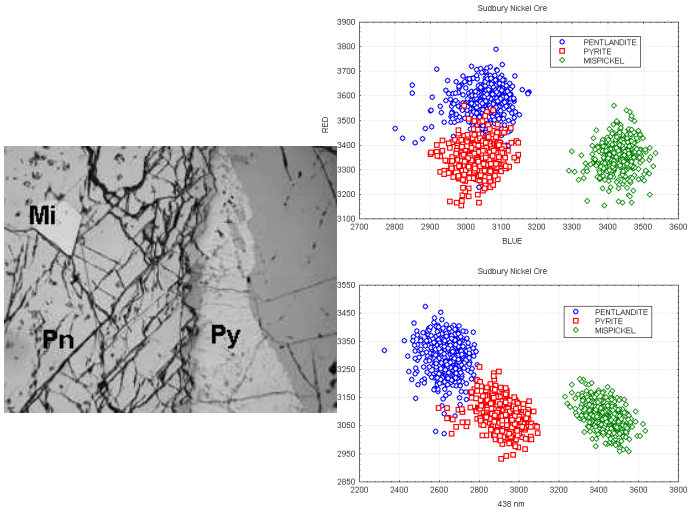

Fig. 1 Optical microscopy of a nickel ore showing Pyrite (Py), Pentlandite (Pn) and Arsenopyrite (Mi). Scatterplots represent selections of 400 representative pixels from each phase when using red vs. blue imaging (upper diagram) or 438 nm vs. 692 nm imaging (lower diagram).

Backscattered electrons imaging under the electron microscope is a very convenient method for quickly discriminating between some major sulphides or sulphides and gangue (Lastra, 1998). It is sensitive to the mean atomic number of a mineral. If an 8 bit depth digitization is used and considering the range of intensities between gangue (typically $\overline{Z} = 10$) and galena ($\overline{Z} = 73{,}2$) or gold ($\overline{Z} = 79$) the spectral resolution is about $\Delta\overline{Z} = 0{,}27$, meaning in practice that magnetite and hematite and to a lesser extent goethite can hardly be discriminated. Not to say that pyrite also falls within the same interval.

Energy Dispersive X-Ray analysis is by far the most accurate and sensitive method. It delivers semi-quantitative chemical analysis of a pear-shaped region hit by the electron beam within a few milliseconds. As a complementary tool to BSE it immediately reveals the difference between pyrite and hematite. However, to differentiate minerals made of the same elements in different proportions, longer integration times are needed (e.g. Pyrite vs. Pyrrhotite). The trilogy of iron oxides is a very subtle case necessitating a light-elements detector for oxygen and exceptional accuracy to distinguish hematite (70 % O) from magnetite (72% O).

Both BSE and EDX imaging modes in scanning electron microscopy cannot reveal a difference between polymorphs or between grains of identical composition but different crystallographic orientations. This requires orientation imaging based on electron diffraction patterns (Adams et al., 1993), which is increasingly used in materials sciences for single phase materials, but still not widespread in geological materials (Prior et al.,1996).

On the contrary, it is common practice to exploit polarization modes in optical microscopy and deduce anisotropy and crystal orientation from light behavior. But, surprisingly again, few efforts have been made to cope with this information quantitatively in optical image analysis (Fueten, 1997). Because specular reflectances are quite close and may vary in practice with composition, polishing compound and even grain size it is worth adding anistropic information to help contrasting magnetite from hematite and goethite.

Finally, imaging of the microporosimetry is also an important topic in iron ore characterization. Here again, due to the limited depth of focus of optical microscopy, excellent contrast is achieved between the polished surface and any default (scratch, cleavage, pore,…) whether imaging under the scanning electron microscope is less favourable except if resin impregnation is used in BSE mode.

3 MULTIRADIAL IMAGING OF OPAQUE MINERALS.

In order to take maximum advantage of optical information, an imaging system combining both multispectral and multiradial (polarised light) capabilities was set up. This system uses a Peltier cooled scientific grade 12 bit CCD camera and a series of interference filters mounted on a filter wheel. Additionally, the microscope has a rotating polar (incident light path) and a rotating analyser (reflected light path). Each image is acquired using a precise background correction protocol and time-averaging of a sequence of images to reduce noise (Pirard, 2004).

Multispectral imaging consists in stacking a series of subsequent images taken at different wavelength in plane polarised light. In order to add information about the anisotropy, another series of images must be taken at different orientations of the polarisation plane with respect to the mineral surface. To gather complete information this should be done at different wavelength under plane polarised light and repeated at these wavelength for analysed light (crossed polars) (Criddle, 1998). Practical considerations make it cumbersome, particularly because image warping (rotation) is necessary to co-register images, so a simplified and approximate procedure has been used in this work.

In transmitted light microscopy, multiradial imaging has been developed by Fueten (1997) by synchronously rotating the polarizer / analyzer instead of the specimen stage. This cannot be achieved in reflected light microscopy because of the semi-reflecting mirror architecture (Criddle, 1998, p21). In other words, polarisation of rays is rotated at oblique angles of incidence and hence the polarisation state varies within the cone of light. If the plane of polarisation of incident light is no longer parallel to the semi-reflecting mirror, this will also induce elliptical polarisation. The result of this being that isotropic minerals will display a false anisotropy and ghost images (secondary glare) might appear (Galopin and Henry, 1972).

For sake of mineral identification in a section relative to one another, it is not mandatory to have the exact measurement of bireflectance / pleochroïsm intensities. The same holds for anistropic reflectance curves (anistropic tints) which are still poorly understood. Hence, in this work, a series of images have been acquired from the same scene when rotating the incident light polar and these were simply stacked together with multispectral information to see whether this could improve discrimination among mineral species.

4 RESULTS

Several characteristic textures of itabirite iron ores were pictured and processed by using supervised classification algorithms available within the MultiSpec software. The richness of multispectral / multiradial information is best shown using false color, alternatively figure 2 presents reflectance and bireflectance images derived from a set of images taken at five different wavelengths and using polarization steps of 15°.

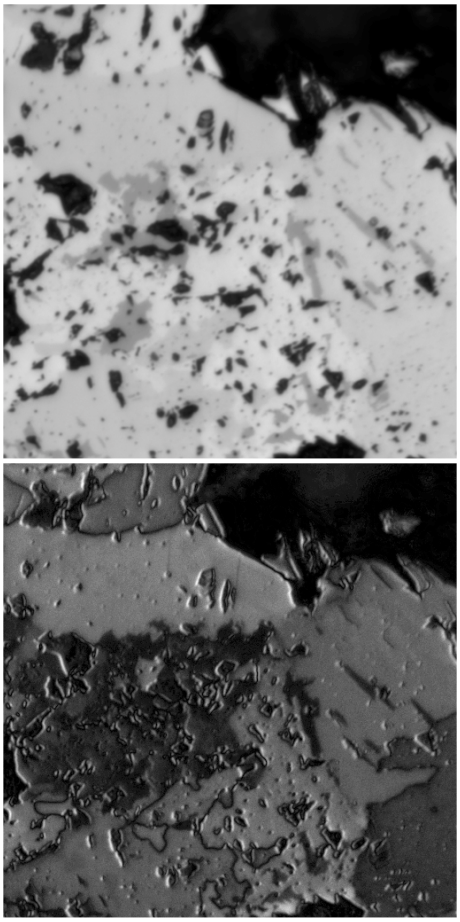

Fig. 2a. Reflectance image at 589 nm displaying porosity (black); magnetite (dark grey) and hematite (light grey) and Fig. 2b. Bireflectance image of the same scene computed from the difference between maximum and minimum reflectance at different orientations of the polarizer.

Clearly, a reflectance image at any wavelength allows for discriminating reasonably well between magnetite and hematite. Average grey levels are in the range of 152 for magnetite and between 186 and 218 for hematite (due to bireflectance). However, by stacking several images while rotating the polarizer, the average grey level difference between the maximum intensity for magnetite and the minimum intensity for hematite increases by about 36 %, thereby improving the robustness of the classification algorithm. If instead of pooling all brightest pixels together, one adds a bireflectance

intensity criterion to the segmentation process, it is straightforward to obtain a mapping of individual hematite crystals (Fig. 3) from which grain size distribution or preferred orientation planes can be derived by image analysis (Launeau et al., 1990).
In practice, despite the non-adequate polarization conditions, isotropic sections of hematite remain relatively constant (0 to 5 grey levels variation). All magnetite crystals show variations of the order of 10 to 15 grey levels, while anisotropic sections of hematite reach bireflectance intensities up to 40 grey levels.

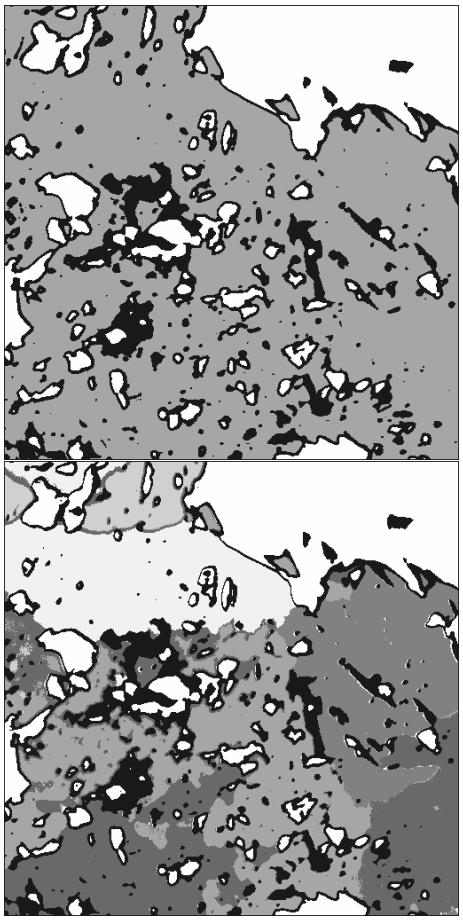

Fig. 3a. Fisher Linear Likelihood classification of hematite (grey); magnetite (black) and pore (white) pixels based on a stack of five spectral and four polarized images.
Fig. 3b. Classification of the same set of images by taking into account a segmentation criterion based on the bireflectance intensity. Magnetite (black), various hematite crystals (from dark to light grey).

5 CONCLUSION

Correct mineral identification is an absolute prerequisite for proper image analysis of ores. This can be achieved by using several imaging methods having their own advantages and disadvantages. Clearly, optical microscopy image analysis still has a promising future if optimal imaging conditions are used and a maximum of information is pooled together in a multivariate classification system.

The exact measurement of optical properties is hard to achieve without major modifications in the microscopical setup, but nevertheless indicative anisotropy is often sufficient to enhance differences and reveal textural informations of interest to the applied mineralogist.
When comparing the relative performance of electron microscopy vs. optical microscopy based instruments, one should no forget the higher productivity of optical instruments. These can possibly inspect much larger sample sets at very high speed, thus putting into practice the famous slogan of stereology and sampling theory claiming "do more less well".
To reach this goal however, additional work has to be performed to improve the automated acquisition of optical image analysis systems using spectral and polar filter sets.

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